Instrumented Indentation

Instrumented indentation is an effective method of characterizing the mechanical properties of thin films and coatings, but its value in coating development and product specifications is limited by the fact that no national or international standard test methods for its use exist. This program seeks to develop standard test methods and materials (SRM's) both for the performance of the test and for the analysis and interpretation of the test results.

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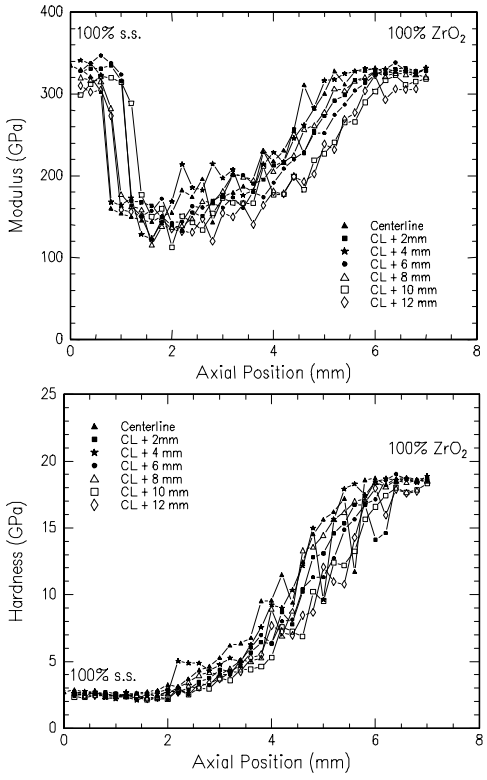
This project deals with the use of instrumented indentation to probe the mechanical properties of coatings and thin films. The indentation machines at NIST permit studies at peak indentation loads ranging from 40• N to over 20 N, using Vickers, Berkovich and spherical diamond and WC-Co indenter tips. The resulting experimental load-displacement curves are analyzed to yield the hardness and Young's modulus of the material probed, as well as the energy absorbed in the indentation process. Since elastic modulus measurements are sensitive to discontinuities in the microstructure, such as cracks and crack-like voids, they are well suited to the study of changes that occur as the result of changing fabrication conditions or post-fabrication treatments. Relatively small volumes of material are probed in each test, permitting local mapping of mechanical property variations and the determination of hardness and elastic modulus for thin films as well as coatings.

The focus of the project is on the development of the technique of instrumented indentation, rather than the application of the technique to particular material systems, although data are taken on specific materials of technological interest (e.g., thermal barrier and wear-resistant coatings), as well as candidates for Standard Reference Materials. International workshops, symposia, and round robin standards tests are organized and executed in an effort to guide the instrumented indentation community toward greater standardization in data analysis, to the expand the range of mechanical property characterization possible with the technique, and to develop physical standards for the technique. In the past year, significant progress has been made within ASTM and ISO on the standardization of the test method. Although these standards deal initially only with the study of bulk materials, they lay the groundwork for later application to coating and thin film testing. NIST is a co-organizer of a large international round robin (VAMAS TWA 22, Project 1), now nearly completed, to study the application of the technique to thin films.

In an unusual application of the technique, studies were done on a series of thick (several mm) functionally graded thermal barrier coatings prepared at the Materials Engineering Laboratory in Tsukuba, Japan, by a spark plasma sintering process. The coating/substrate system consisted of a pure 410 stainless steel base, a pure yttria-stabilized zirconia top coat and nine intermediate layers of mixed composition to transition smoothly from base to top coat. Polished cross-sections of the coating were indented to measure hardness and Young's modulus through the coating thickness. The results, Figure 1 beloww, showed that although the hardness increased monotonically with zirconia content, the modulus of several of the intermediate layers was anomalously low. This was later shown to be due to insufficient sintering in those layers, a problem that was not recognized prior to the indentation work.

Figure 1: Hardness and modulus through the thickness (axial position) of a graded stainless steel/zirconia coating, for several distances from the central axis of the specimen.



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